California Environmental Protection Agency

Air Resources Board

Draft

Protocol for the Determination of the Permeation Rate from Preconditioned Phase II California Reformulated Certification Fuel Versus Preconditioned Commercial Pump Fuel through High-Density Polyethylene Containers

Engineering and Certification Branch
Monitoring and Laboratory Division

Project No.
T-00-070 Permeation Study

Date: December 5, 2000

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Protocol for the Determination of the Permeation Rate from
Preconditioned Phase II California Reformulated Certification Fuel
Versus Preconditioned Commercial Pump Fuel through
High-Density Polyethylene Containers

I. Introduction

This protocol will describe a study to investigate the permeation rates of gasoline containers preconditioned with Phase II California Reformulated Certification (CERT) fuel versus commercial (pump) fuel. This testing will be done to verify similar testing conducted by Phillips (July 18, 2000 Memorandum, Raboin to Bloudoff and September 15, 2000 Memorandum, Dillard to Lew). The study will follow the procedures described in the Air Resources Board (ARB) Test Method 513 "Determination of Permeation Rate for Spill-Proof Systems" (July 6, 2000, see Attachment C) with the exception of using CERT fuel for preconditioning. The gasoline containers being tested have been treated with 1) Selar®, 2) fluorination, and 3) sulfonation. Untreated containers will also be tested for comparison.

Phillips conducted a 13 week test primarily intended to compare the permeation rates of various Marlex® resins. Included in this test was a comparison of the preconditioning method in the California Air Resources Board (CARB) Test Method 513 with that of ASTM PS 91 (see Attachment D). CARB Test Method 513 uses pump gasoline in preconditioning followed by CERT fuel for the permeation test. The ASTM method PS 91 uses a single filling of CERT fuel for preconditioning and the permeation test. Phillips results indicate that during the first couple of weeks the preconditioned containers with the pump fuel had a higher permeation rate versus the containers with preconditioned CERT fuel. After the first couple of weeks the permeation rate reached steady state for both methods. The difference after the first couple of weeks is both methods reached steady state, but the preconditioned containers in pump fuel were noticeably higher in the permeation rate.

The containers will be tested using the Sealed Housing for Evaporative Determination (SHED) at the Air Resources Board El Monte facility. The primary purpose of the SHED is to maintain a temperature controlled environment that will provide the necessary temperature profile, as outlined in Attachment A.

For preconditioning half the containers will be filled with commercial fuel and half with CERT fuel. The containers will then be placed in the SHED for a 72-hour cycle to allow equilibration of the permeation rate. At the end of the 72-hour cycle, a 24-hour cycle for 10 consecutive days will be conducted with the weight measured after every cycle. At the end of the 10th day the containers will be emptied, blown dry with air and weighed. The average permeation rate will be calculated based on results of the 10 day test.

II. Description of Fuel Containers

Several containers from Wedco Moulded Products and Scepter Corporation were chosen for testing. Wedco submitted five each of 1.25-gallon, 2.5-gallon and 5.0-gallon containers that are fluorinated at level 5 (higher level of fluorination represents higher resistance to permeation). Wedco also submitted five each of 1.25-gallon, 2.5-gallon and 5.0-gallon sulfonated and untreated containers. Scepter submitted five each of 1-gallon, 2-gallon and 5-gallon containers treated with Selar® at 8% (Percentage of Selar® resin in the HDPE).

III. Test Procedures

A. Fuel

The CERT and pump fuel used will be taken from the El Monte facility. At the time of testing we will ask the fuel coordinator in El Monte for a current value of the CERT fuel density that will be used in calculating the permeation rate. If a current value is not available, then a density of 2,793.3 grams/gallon will be used, as measured from CERT fuel used in previous tests. We will also attain other data necessary for comparison between CERT and pump fuel (e.g. RVP, etc.). All containers will be filled the same day and at the same dispensing terminal. Containers that are the same size will be filled with the same volume of fuel to eliminate the potential for permeation rate variability due to different fuel volumes.

B. Container Labeling

Container identification will consist of several different styles depending on the treatment and fuel being used. An identification of a container will include the manufacturer, container size, fuel type and a sequence number for containers of the same type. The container identification will be permanently marked on the container and performed before the durability test as described in section C. Examples of sample identifications with description of the different styles is listed below:

Untreated **W1-01C**	Fluorinated **T5L4-01P**	Sulfonated **M2S-01C**	Selar **W1S8-01P**
W - Mfg. 1 - Size (gallon) 01 - Sequence No. C or P - Fuel Type	T - Mfg. 5 - Size (gallon) L4 - Fluorination Level 01 - Sequence No. C or P - Fuel Type	M - Mfg. 2 - Size (gallon) S - Sulfonation 01 - Sequence No. C or P - Fuel Type	W - Mfg. 1 - Size (gallon) S8 - Selar Percentage 01 - Sequence No. C or P - Fuel Type
Fuel Type: "C" - CERT, "P" - Pump			

Container labels will include information on date of first day of testing, project name, project number, sample identification and name of person(s) who prepared the container for testing. Labels will be laminated, to prevent tear and water damage, and required information mentioned above will be written on the laminated label with a permanent marker at the time of testing.

C. Durability Test

Each container will undergo 1000 cycles of vacuum at 2 inchesHg (0.98 psi) and pressure at 5.0 psi. The air temperature supplied will be at 120°F. All container identification information will be entered on a PC including the technician performing the tests.

D. Container Wall Leak Check

A leak check of the container walls and seams will minimize the possibility of vapor loss due to manufacturing defects and will verify integrity during testing. The first step is to pressurize the container, with air at 140°F, to approximately 20 psi and hold. Then using a water bath, submerge the pressurized container completely for two minutes and check for air bubbles. The check consists of rotating the container around completely so that all walls face upward to allow bubbles, if any, to escape. Leaks around the cap are allowed because the opening will be sealed with a high-density polyethylene (HDPE) disc before testing and leak checked as described in section F. If leaks occur from the container walls, then using a permanent marker circle the area(s) where it leaks and mark the container as “Wall Leak.” The data sheet will have a pass/fail area to record results for this check.

E. Preconditioning

For preconditioning, half of the containers will be filled with CERT fuel and the other half with pump fuel. The fuel type will be identified as specified in the labeling requirements. At a minimum each container shall be filled to its rated capacity. Each container shall undergo a minimum soaking duration of 30 days stored at ambient temperature and pressure. A record will be kept for actual days of soaking and fuel type used for each container.

F. Preliminary Test Activities

After a minimum preconditioning period of 30 days all containers will be emptied and immediately filled with CERT fuel. All equal volume containers will be filled to approximately the same volume (rated capacity) of fuel and the volume recorded. The container opening will be sealed by fusion welding an HDPE disc on top. Melting its own plastic or using an HDPE disc will seal the small secondary port (if included on the container). The containers will then be placed in the SHED at 110°F for approximately two hours. Positive internal pressure is

observed by container swelling. After heating, all containers will be leak checked by submerging the containers under water and looking for air bubbles around the HDPE disc(s).

G. Balance Requirements

The model LP 16000S Sartorius balance used for this test is located next to the SHED and will not be moved during the test after calibration at its location. The readability (tolerance) is ±0.1 grams and weighing capacity is 16,000 grams. The building air-conditioning units must be turned off and the shop doors closed before weighing the containers. Wind currents may cause variability in the balance reading. At the start of every day wipe the top surface of the balance and perform the internal calibration check by weighing a 1000-gram standard weight. The standard weight is traceable to the National Institute of Standards and Technology (NIST). Weigh the 1000-gram standard after every 10 measurements and record the result on the data sheet (see Attachment B). When using the 1000-gram standard the balance must read between 999.9 and 1000.1 grams to pass the calibration checks. If the reading is outside of this tolerance then the balance is re-tared and only the previous 10 measurements will need to be re-measured.

H. Permeation Test

All containers will be wiped with a cloth to remove any external debris before weighing. Using the balance, the containers will be weighed and initial weight recorded. When containers are placed in the SHED, they are not to touch each other. The containers will undergo an initial 72-hour cycle, which on a daily basis uses the 24-hour temperature profile as shown on Attachment A. At the end of the 72-hour cycle the containers begin a 24-hour cycle for 10 consecutive days with the weight measured after every cycle and recorded. The temperature profile used is as shown on Attachment A.

I. Post Test Activities

After permeation tests are completed, the containers will be placed in the SHED at 110°F for approximately two hours. Positive internal pressure is observed by container swelling. After heating, all containers will be leak checked by submerging the containers under water and looking for air bubbles around the HDPE disc(s). Then the containers will be opened and fuel emptied into 55-gallon drums. All parts associated with each container after opening will be kept together for post weight measurement. After containers are empty, dry the inside and outside of container with compressed air. Weigh each container with its associated parts and record weight (tare weight) on data sheet. The tare weight will be used in calculating the permeation rate. All containers will then be stored in the flammable storage cabinets in El Monte for future use.

IV. Quality Assurance

Quality Control consists of having half the containers filled with CERT fuel and the other half with commercial pump fuel (only during preconditioning). This will allow for comparison of any differences in the permeation rate during testing.

All containers will be filled as specified in the Preliminary Test Activities. Containers filled to the same volume will reduce the variability of the permeation rate due to different volumes of fuel per container.

A control sample will also be included during this test and weight recorded. This consists of a metal gasoline container that is sealed with a non-permeating epoxy. The epoxy will seal the container ports and the container will be leak checked by submerging into a water bath. The preconditioning and durability test will not be performed for this container. The purpose of this non-permeating sample is to act as a control for the determination of measurement variability over the course of the study.

V. Permeation Rate Calculation

The Test Method 513 permeation rate formula will be used for this test. A current density (g/gal) number will be determined at the time of testing in El Monte.

VI. Personnel

ARB personnel will consist of Oscar Lopez (Project Engineer) and Instrument Technicians from the Testing Section.

VII. Safety Precautions

When emptying or filling containers with fuel a certified fuel mask and goggles will be used. Never fill containers to the very top of opening; heat gun used for sealing may come in contact with fuel. Always work in ventilated areas when working around fuel.

Attachment A

SHED Temperature Test Profile

SHED Temperature Test Profile

Hour	Minute	Temperature (°F)
0	0	65.0
1	60	66.6
2	120	72.6
3	180	80.3
4	240	86.1
5	300	90.6
6	360	94.6
7	420	98.1
8	480	101.2
9	540	103.4
10	600	104.9
11	660	105.0
12	720	104.2
13	780	101.1
14	840	95.3
15	900	88.8
16	960	84.4
17	1020	80.8
18	1080	77.8
19	1140	75.3
20	1200	72.0
21	1260	70.0
22	1320	68.2
23	1380	66.5
24	1440	65.0

Attachment B

Data Sheet

SHED PERMEATION TEST
DATA SHEET

Container I.D. _____ Container Volume: _____

Manufacturer: _____

Tested By: _____

Wall Leak Check (pass/fail): _____

Seal Leak Check (pass/fail): Pre - _____ Post - _____

Tare Weight - W_t (grams): _____

Preconditioning Duration (days): _____

	DATE		START	INITIAL WEIGHT	FINAL WEIGHT	WEIGHT LOSS	
	START	STOP	TIME	W_i (grams)	W_f (grams)	W_l (grams)	STD
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							

NOTES: _____

Attachment C

Test Method 513
Determination of Permeation Rate for Spill-Proof Systems

Attachment D

ASTM PS 91-00
Provisional Standard Specification for Portable Gasoline And Kerosine Spill Resistant Fueling Systems for Consumer Use